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Celluloid in Ammunition:
Part 2: Comparison of 'Firmoid' with Celluloid and the Incidence of Embrittlement

G. W. Harding
T. K. Overton

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Celluloid in Ammunition: Part 2: Comparison of 'Firmoid' with Celluloid and the Incidence of Embrittlement

by

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Celluloid in Ammonium
Part 2: Comparison of 'Firmoid' with Celluloid and the Incidence of Embrittlement

by

G.W. Harding and T.K. Overton

References: XR.320/34, M(R) 5/1

Any communications concerning this Report should be addressed to D.M.X.R.D. Ministry of Supply, Shell Mex House, Strand, London, W.C.2.

1. SUMMARY

'Firmoid', a proprietary spray-deposited celluloid specially suited to the fabrication of items of difficult shape, has been compared with celluloid by heat-ageing trials at 60°C, both in the presence and in the absence of a double-base propellant.

It has been shown that physico-chemically both materials behave similarly, absorbing nitroglycerine in exchange for camphor and losing strength, but of the two 'Firmoid' remains much less flexible throughout. However, in cases where, for reasons of fabrication, 'Firmoid' is preferred to celluloid, and its lack of flexibility is not an insuperable objection, it can be expected to give a useful though possibly a more limited service life. Continuation of the trial until the onset of embrittlement showed that this phenomenon is associated with degradation of the cellulose nitrate ingredient of celluloid. It occurs if the molecular weight of the polymer falls below a critical figure of about 16,000.

2. INTRODUCTION

Part 1 of this Report (1) referred to the tendency of celluloid to become brittle on ageing and also to the association of embrittlement with the presence of double-base propellant. The results of this work showed that the physico--chemical behaviour of 'Firmoid', a spray-deposited celluloid, is similar to that of sheet celluloid. Thus it absorbs nitroglycerine from double-base propellant and loses camphor to the propellant at the same time. It is however always much less flexible than celluloid, i.e. it is more readily broken when bent round suitable small mandrels, and cannot therefore be considered a suitable alternative to celluloid unless it has over-riding advantages in some other direction.

Forward-venting primers and igniters currently being developed require magazine liners of greater size and more intricate shape than hitherto and it has been found exceedingly difficult to fabricate such items from sheet celluloid. "Firmoid" on the other hand can be readily formed in to the required shapes.

/Knowledge .....

Knowledge of the ageing characteristics of 'Firmoid' in comparison with those of sheet celluloid therefore became an essential requirement, especially as the service life of the celluloid is often relatively limited, for reasons not understood at the time this work was started.

This limited life is emphasised in a recent report on the outdoor exposure of celluloids in the tropics (2), where it was observed that the onset of brittleness was rapid, especially in the desert, i.e. in a few months.

3. OBJECT

The purpose of this work was twofold. The first aim was to compare 'Firmoid' with celluloid over an extended period of ageing by various physical and chemical tests in order to determine whether 'Firmoid' was likely to have a useful life. The second aim, which was achieved by extending the trials to the onset of brittleness in the materials, was to determine the cause for this brittleness in the hope that some steps could be taken to minimise its incidence in Service.

4. EXPERIMENTAL

4.1 Materials

The materials used in this trial, celluloid, 'Firmoid' and propellant WM, were as described in Part 1 of this Report.

4.2 Trial

Several strips of both 'Firmoid' and celluloid were placed between sheets of propellant WM, sandwich fashion, as in the previous trial and were subjected, in sealed containers, to heat-ageing at 60°C. Similar strips, acting as controls, were placed between sheets of cardboard inside sealed containers and subjected to the same ageing conditions. Specimens were withdrawn periodically until the trial was terminated at the end of 956 days.

4.3 Testing

Details of changes in both the appearance and the weight of the films were noted and analytical and flexibility tests were carried out as described in Part 1 of this Report.

In addition, determinations were made of the molecular weights of portions of cellulose nitrate polymer extracted from films which had undergone 409 days storage and longer. These were compared with similar determinations of cellulose nitrates extracted from the original materials.

The cellulose nitrate samples were obtained as residues from the solvent extraction of specimens of film for infra-red analysis. After the polymers had been dried they were dissolved in acetone and viscosity determinations were made at various concentrations at 25°C, using a modified Ubbelohde viscometer.

/Molecular .....

Molecular weights were then deduced by the method given by Mark and Tobolsky (3) from a knowledge of the "intrinsic viscosity" and by application of the modified Staulinger equation, thus:

$$[\eta] = \lim_{C \to 0} \frac{\eta - \eta_o}{\eta_o C}$$

where $[\eta]$ = intrinsic viscosity

η = viscosity of solution

η_o = viscosity of solvent

C = concentration in g/100 ml. of solution

and $[\eta] = KM^{\alpha}$

where M is molecular weight and K and α are constants. For cellulose nitrate in acetone $K = 0.38 \times 10^{-4}$ and $\alpha = 1$ (3).

The tensile strengths of strips of 'Firmoid' and celluloid were also determined both before and after ageing for 956 days. The tests were carried out on a Baldwin Universal Testing Machine with rate of separation of the grips of 1 inch per minute.

5. RESULTS

There was little change in the appearance of the control specimens of either celluloid or 'Firmoid' until a late stage in the trial, when some slight yellowing was noticed.

In contact with propellant WM the celluloid became yellow almost immediately and showed considerable crinkling. As the trial progressed the colour changed from yellow to greenish yellow and finally to a dark brown. 'Firmoid' showed the same colour changes but did not crinkle.

The results are given in Tables 1, 2 and 3 and Figure 1. The control specimens i.e. those aged by themselves, will be dealt with first.

5.1 Controls

The molecular weight determinations (Table 1) show that the cellulose nitrates from both the celluloid and the 'Firmoid' control specimens were degraded to a similar extent. At the end of the trial this degradation, although considerable, had not seriously affected the physical properties of the materials. The tensile strengths of both the celluloid and the 'Firmoid' were in fact higher at the end of the trial than at the beginning (Table 2): this is probably due to the loss of plasticiser and/or residual solvent.

The weight loss (Table 3) tended to increase with ageing, although rather irregularly.

The weight lost by the control specimens of 'Firmoid' was slightly greater than that lost by the celluloid controls. It is also seen (Table 3) that the camphor content of both materials fell sharply early in the trial and then tended to level off, but that of the 'Firmoid', initially rather lower than that of the celluloid, finally fell much further than did that of the celluloid.

/It .....

It might be expected that the "weight loss plus camphor content" would be fairly constant, on the assumption that weight changes are wholly due to loss of camphor. Some variation was observed however. This is probably because the infra-red method of determination of camphor is only approximate.

The results of the flexibility tests on the control samples (Table 3) show that, within the limits of the experiment, celluloid was still as flexible at the end of the trial as it was originally, whereas the 'Firmoid' had become less flexible.

5.2 Specimens Aged in Contact with Propellant WM

Molecular weight determinations (Table 1) revealed a degradation of the cellulose nitrate in both celluloid and 'Firmoid' after contact with propellant. The degree of degradation was similar in the two materials, but was greater than in the control specimens. Table 2 shows that, in contrast to the controls, the specimens that had been in contact with the propellant were seriously affected physically, both celluloid and 'Firmoid' having much lower tensile strengths than either the controls or the unaged specimens.

Both materials showed a considerable increase in weight (Table 3) in a very short time due to high absorption of nitroglycerine, notwithstanding that a large part of the camphor was lost, at the same time, from both celluloid and 'Firmoid' to the propellant.

Both materials became softer owing to the absorption of nitroglycerine. This was indicated by the flexibility test in which 'Firmoid' immediately became more flexible and could be bent on itself. This test was not stringent enough to indicate any significant difference between the two materials. After 184 days on trial the 'Firmoid' appeared to become less flexible although it was still highly plasticised. By the end of the trial it had become brittle. This trend towards brittleness was also apparent in the celluloid at the end of the trial when it cracked on being bent on itself, although it was still capable of being bent round the 1/16 inch mandrel.

6. DISCUSSION

If allowance is made for the considerably greater initial flexibility of celluloid it is seen that the performances of celluloid and 'Firmoid' under the conditions of the trial are similar, both chemically and physically.

It may be inferred that both materials should have a long like where they do not come into contact with propellant. Where this contact does occur, a shortened but still useful life should be possible provided that the swelling and softening due to absorption of nitroglycerine, changes which are only partially offset by migration of camphor, are not unacceptably large. These effects occur almost immediately as shown in Fig.1. If therefore a component made of celluloid is still functioning satisfactorily after about a week it should continue to be effective for some time.

6.1 Embrittlement of Celluloid and its Possible Prevention

The doubts expressed in the past about the serviceability of celluloid, due to poor ageing characteristics, and the phenomenon of embrittlement in the presence of double-base propellant may now be examined in the light of the trial results.

/A first .....

A first theory is that embrittlement is associated with loss of plasticiser. This must be ruled out as a major factor however, for the reasons (i) that sheet celluloid retains its flexibility at the end of 956 days at 60°C, despite the loss of about one quarter of its camphor, and (ii) that in contact with WM propellant, after absorbing nitroglycerine, which initially produces a powerful plasticising effect, it eventually embrittles within 956 days, even though the plasticiser content remains almost unchanged, from 24 hours onwards.

A second possibility, that embrittlement is associated with the breakdown of the cellulose nitrate molecule, fits in much better with the facts.

The experiments showed that the cellulose nitrates from the celluloid and 'Firmoid' as received had molecular weights of 61,700 and 48,700 respectively. The materials were not brittle, subject to the limitation imposed by the physical characteristics of 'Firmoid'. After 956 days at 60°C they showed no loss of tensile strength and the celluloid was still flexible, but the 'Firmoid' had become rather brittle. The cellulose nitrates from these aged specimens had molecular weights of 24,500 and 20,300 respectively, indicating a considerable amount of degradation, although without signs of serious embrittlement.

On the other hand, both celluloid and 'Firmoid', after contact with propellant WM under the same conditions, had very low tensile strengths. They were either beginning to show signs of embrittlement (celluloid) or had become embrittled ('Firmoid'), and the molecular weights of the cellulose nitrates were 13,200 and 14,100 respectively.

It therefore appears that serious embrittlement of celluloid occurs when the molecular weight of the polymer is reduced to between 14,000 and 20,000. This conclusion is borne out by the experience of other workers. It has been stated (4) for instance, that celluloid made from $\frac{1}{2}$-second cellulose nitrate is quickly embrittled, whereas celluloid made with 15-second cellulose nitrate is not, and is quite satisfactory in use.

Miles (6) quotes $\frac{1}{2}$-second cellulose nitrate as having degree of polymerisation (DP) of 63, and 12.5-second cellulose nitrate as having a DP of 150, where DP is the number of pyranose units in the chain. These materials therefore have molecular weights of about 16,000 and 38,000 respectively. 15-second cellulose nitrate would have a molecular weight higher than 38,000. It is not surprising therefore that celluloid made with $\frac{1}{2}$-second cellulose nitrate is either brittle or becomes brittle very quickly.

This experimental work is also in agreement with the further observations of Miles (7) who states that the tensile strength of cellulose nitrate films is independent of the viscosity of the polymer until the intrinsic viscosity is in the neighbourhood of 0.07, and that this value corresponds to the viscosity of the $\frac{1}{2}$-second cellulose nitrate with a DP of about 60.

(Note: The expressions "$\frac{1}{2}$-second" and "15-second" (5) are technical terms relating to the viscosity of concentrated solutions of cellulose nitrate. They are the times a standard steel ball takes to fall through 10 inches of a solution containing 12 grammes of cellulose nitrate in 100 grammes of solution. The longer the time of fall the higher the molecular weight of the cellulose nitrate).

/It is .....

It is apparent from the molecular weight determinations that celluloid degrades right from the beginning of the trial, and embrittlement will occur when sufficient time has elapsed for the molecular weight of the cellulose nitrate to fall below a critical value of about 16,000. The time to embrittlement will therefore be largely dependent on two things, first the molecular weight of the original cellulose nitrate (the higher the molecular weight the longer it will be before embrittlement occurs), and secondly the rate of breakdown of the molecular chain.

The mechanism of breakdown has not been examined in this work. In the absence of propellant it would be reasonable to suppose that oxidative degradation, together with formation of oxides of nitrogen, causes auto-catalytic degradation (8). In the presence of propellant the degradation is accelerated; this is possibly due to catalysis of the degradation reaction by further small quantities of nitrogen oxides formed by the propellant.

So far as is known, it appears that, apart from the stabilising process which is a part of the preparation of the cellulose nitrate, no other means, e.g. the use of antioxidants, are employed commercially to stabilise celluloid. It is possible therefore that the presence of an antioxidant in celluloid might delay its degradation, or that an acid acceptor would have a beneficial effect, by reacting with any nitrogen oxides evolved by the celluloid, compare for instance the use of carbamite in cordite. However, it was shown in Part 1 that carbamite was absorbed by celluloid so it is possible that an acid acceptor may have been present in the propellant WM trial.

7. CONCLUSIONS

Celluloid and 'Firmoid' made from cellulose nitrate of sufficiently high molecular weight will have long periods of useful life whether or not in contact with propellant WM. Their lives will be longer in the absence of propellant.

Celluloid is better than 'Firmoid' under comparable conditions. The occurrence of brittleness must be ascribed in both cases to the degradation of the cellulose nitrate polymer. This degradation proceeds continuously under suitable conditions and the onset of brittleness coincides with a fall in molecular weight to a critical figure of about 16,000.

Owing to the brittleness inherent in its cellular structure, 'Firmoid' should not be considered as a replacement for celluloid unless fabrication of components from celluloid is not feasible.

8. ACKNOWLEDGEMENTS

The authors wish to acknowledge the help of E.R.D.E. in supplying the propellant and for carrying out the analytical work.

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5. Miles, F.D., Cellulose Nitrate, Oliver and Boyd, London and Edinburgh, 1955, p.135.

6. Ibid, p.138.

7. Ibid, p.143.

8. Ibid, p.103.

/TABLE 1 .....

TABLE 1

Change in Molecular Weight of Celluloid and 'Firmoid' after ageing at 60°C both alone (control) and in contact with propellant WM

Material / Period of Ageing (days)	Celluloid				'Firmoid'			
	Control		With Propellant		Control		With Propellant	
	I.V.	M.W.	I.V.	M.W.	I.V.	M.W.	I.V.	M.W.
0	2.32	61,700	2.32	61,700	1.85	48,700	1.85	48,700
409	1.24	32,600	0.94	24,700	1.41	37,100	1.09	28,700
556	1.26	33,200	0.85	22,400	1.10	29,000	0.79	20,800
956	0.93	24,500	0.50	13,200	0.77	20,300	0.535	14,100

Notes. I.V. ≡ Intrinsic viscosity
M.W. ≡ Molecular weight.

TABLE 2

Change in Strength Properties of Celluloid and 'Firmoid' after ageing at 60°C both alone (control) and in contact with propellant WM

Material / Period of Ageing (days)	Celluloid				'Firmoid'			
	Control		With Propellant		Control		With Propellant	
	T.S. lb/in.^{2}	E.B. (%)	T.S. lb/in.^{2}	E.B. (%)	T.S. lb/in.^{2}	E.B. (%)	T.S. lb/in.^{2}	E.B. (%)
0	8550	18.7	8550	18.7	3770	8.4	3770	8.4
956	11000	16.9	1000	9.5	5460	13.9	1650	9.5

Notes. T.S. ≡ Tensile strength
E.B. ≡ Elongation at break.

/TABLE 3 .....

(1)

Comparison of 'Firmoid'

A. In Air
B. In Con

Contact	Control					
Period (days)	Weight Loss (% of original weight)		Camphor Content (% of original weight)		*Flexibility (mandrel size) (in.)	
	Celluloid	'Firmoid'	Celluloid	'Firmoid	Celluloid	'Firmoid'
0	-	-	19	17	0	5/64
1	3.3	2.7	18.4	12.6	0	3/32
4	1.1	2.4	16.8	15.6	0	3/32
8	1.7	2.8	15.7	15.6	0	$\frac{1}{8}$
15	3.5	3.4	16.4	14.5	0	variable 1/16 and 3/16
29	2.7	3.8	15.6	13.5	0	5/64
57	2.0	3.3	16.7	14.5	0	5/64
93	2.4	4.0	16.6	13.4	0	3/16
184	3.9	4.9	13.0	12.4	0	3/16
291	4.7	6.2	15.7	14.2	0	$\frac{1}{8}$
409	5.3	5.3	14.2	15.4	0	3/32
556	4.2	5.3	15.2	13.7	0	3/32
956	3.6	4.9	14.9	9.0	0	$\frac{1}{8}$

Notes. * Figures in these columns indicate the diameter of the

0 indicates that specimens were bent on themselves with
bending round a 1/16-inch mandrel, the smallest size

**F indicates that specimen cracked when bent round $\frac{1}{2}$ i

TABLE 3

with Celluloid on ageing at 60°C

(in Sealed Container)
tact with Propellant WM

Propellant WM							
Weight Gain (% of original weight)		Camphor Content (% of original weight)		Nitroglycerine Content (% of original weight)		*Flexibility (mandrel size in.)	
Celluloid	'Firmoid'	Celluoid	'Firmoid'	Celluloid	'Firmoid'	Celluloid	'Firmoid'
-	-	19	17	-	-	0	5/64
14.2	13.9	3.8	6.7	28.6	23.9	0	0
18.6	17.4	3.0	4.0	33.2	30.5	0	0
17.5	16.6	2.4	2.3	32.9	30.9	0	0
17.0	16.5	1.6	1.9	32.8	31.5	0	0
16.8	16.3	1.5	1.6	33.3	31.4	0	0
17.3	17.3	1.4	1.4	34.0	32.8	0	0
15.2	15.6	1.5	1.5	31.3	31.3	0	0
15.7	16.3	3.5	2.3	32.4	30.2	0	3/32
15.6	16.5	1.7	1.7	31.9	31.1	0	5/64
16.1	17.2	1.9	1.9	31.1	31.5	0	1/16
15.3	15.9	1.0	0.9	34.5	33.1	0	3/32
13.9	14.4	1.8	1.8	31.1	30.4	1/16	½ **F

smallest mandrel round which a specimen could be bent without cracking.

out cracking. This last test was not tried until specimens had withstood
available.

nch mandrel.

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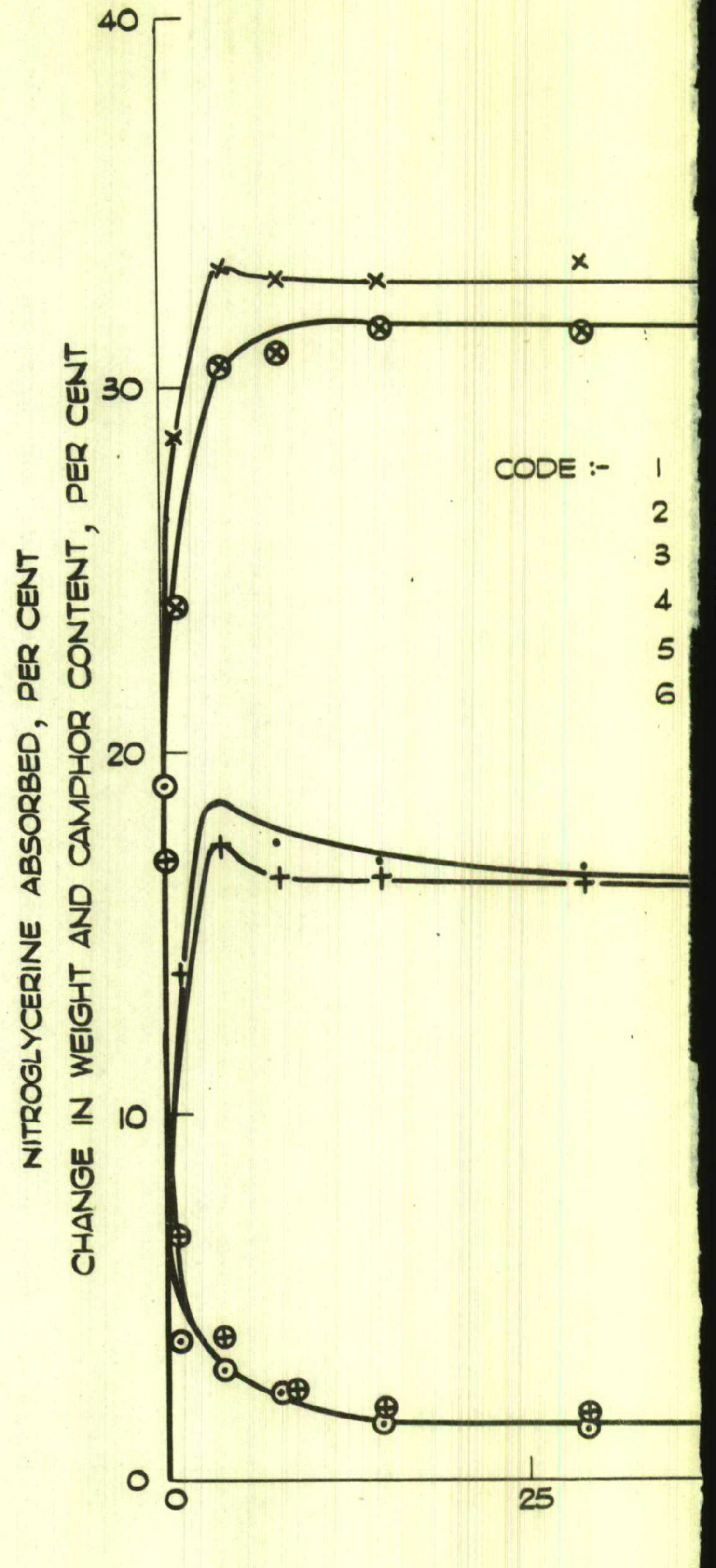
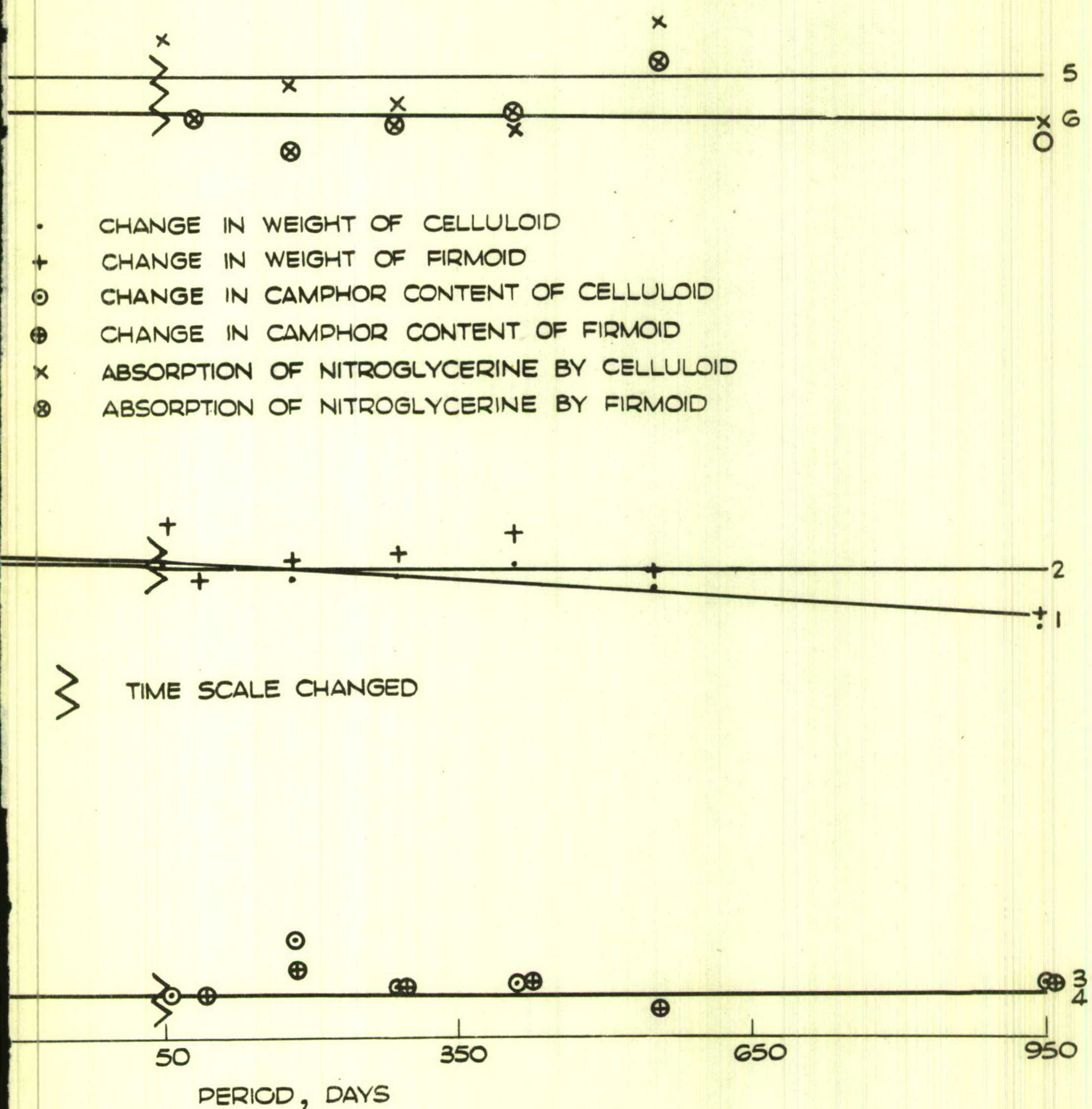


EFFECT OF PROPELLANT



WM ON CELLULOID AND FIRMOID. FIG. 1.

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It has been shown that physico-chemically both materials behave similarly, absorbing nitroglycerine in exchange for camphor and losing strength, but of the two 'Firmoid' remains much less flexible throughout. However, in cases where, for reasons of fabrication, 'Firmoid' is preferred to celluloid, and its lack of flexibility is not an insuperable objection, it can be expected to give a useful though possibly a more limited service life. Continuation of the trial until the onset of embrittlement showed that this phenomenon is associated with degradation of the cellulose nitrate ingredient of celluloid. It occurs if the molecular weight of the polymer falls below a critical figure of about 16,000.

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It has been shown that physico-chemically both materials behave similarly, absorbing nitrglycerine in exchange for camphor and losing strength, but of the two 'Firmoid' remains much less flexible throughout. However, in cases where, for reasons of fabrication, 'Firmoid' is preferred to celluloid, and its lack of flexibility is not an insuperable objection, it can be expected to give a useful though possibly a more limited service life. Continuation of the trial until the onset of embrittlement showed that this phenomenon is associated with degradation of the cellulose nitrate ingredient of celluloid. It occurs if the molecular weight of the polymer falls below a critical figure of about 16,000.

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Celluloid in Ammunition: Part 2: Comparison of 'Firmoid' with Celluloid and the Incidence of Embrittlement

G.W. Harding and T.K. Overton — January, 1959

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